CLASS-1
Physical demonstration of Silicon wafers and surface cleaning procedure for making of MOS cap. Device platform

Exp. No. 1: Studies on Silicon Wafer Cleaning

Objective:

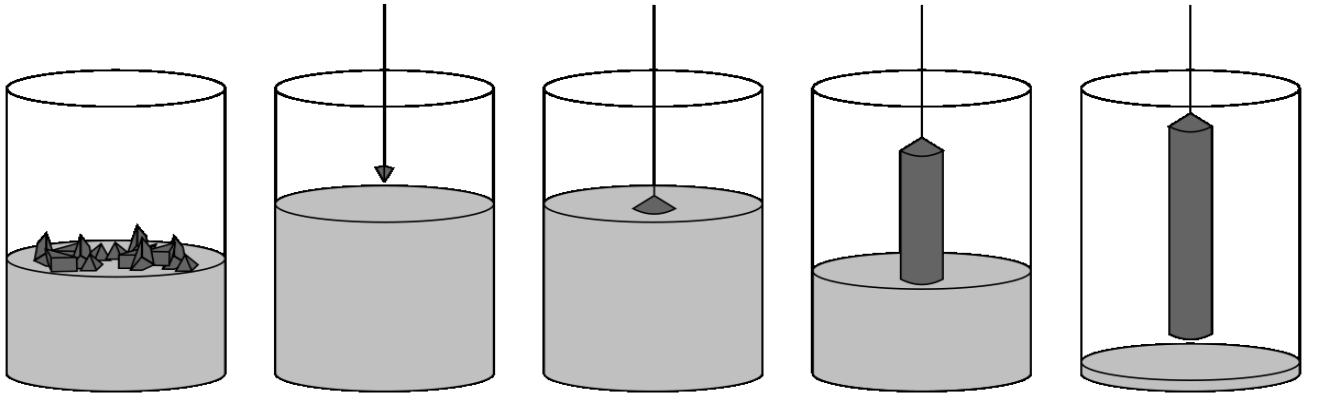
- To learn how many types of silicon wafers are available.
- To learn how to handle silicon wafers.
- To clean the surface of silicon wafer before processing.

Theory:

- Wafers are thin substrate on which micro-fabrication is done.

Specification of Si wafer

Parameters	Possible value
Size	1", 2", 3", 4", 6", 8", 12", 14"
Orientation	<100>, <111>
Grade	Prime > Solar> Test> Mechanical
Type	n type or p type
Method	Float Zone (FZ), Czochralski (CZ) or Microcrystalline
Thickness	200 to 2000µm
Resistivity	< 0.005 Ω cm to 10^5 Ω cm
Finish	Single Side Polished (SSP) or Double Side Polished (DSP)



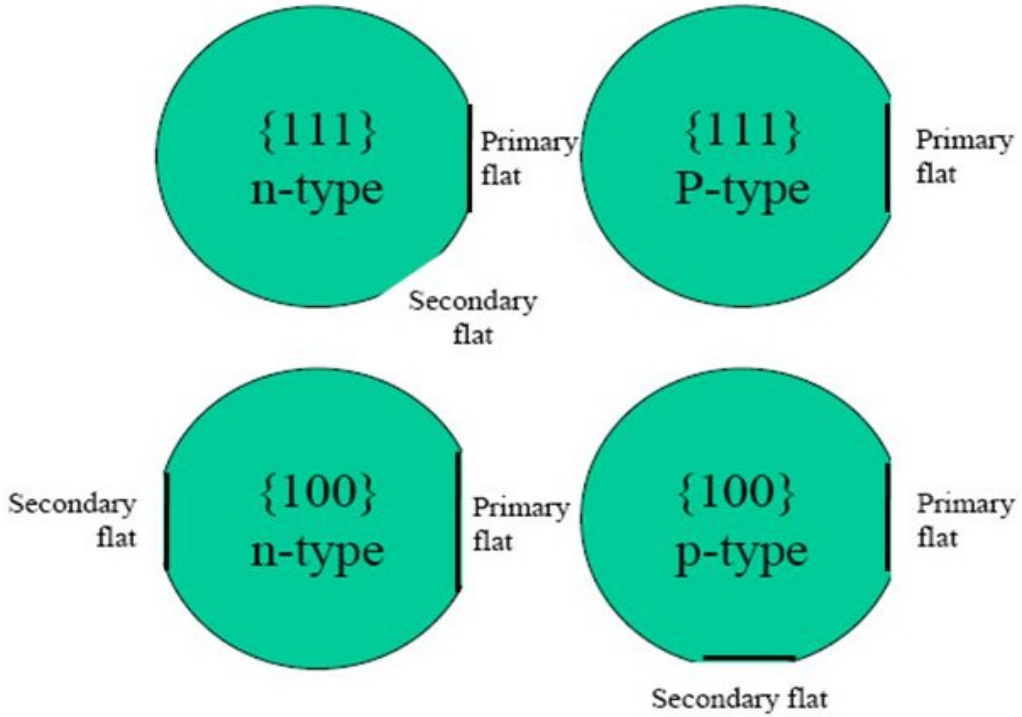
Melting of polysilicon, doping

Introduction of the seed crystal

Beginning of the crystal growth

Crystal pulling

Formed crystal with a residue of melted silicon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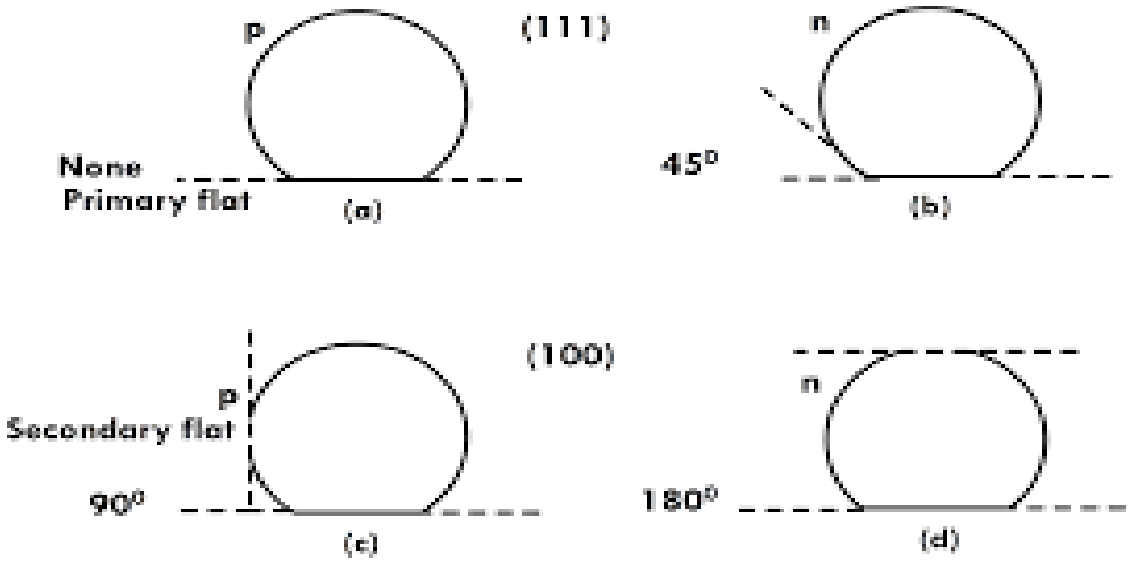
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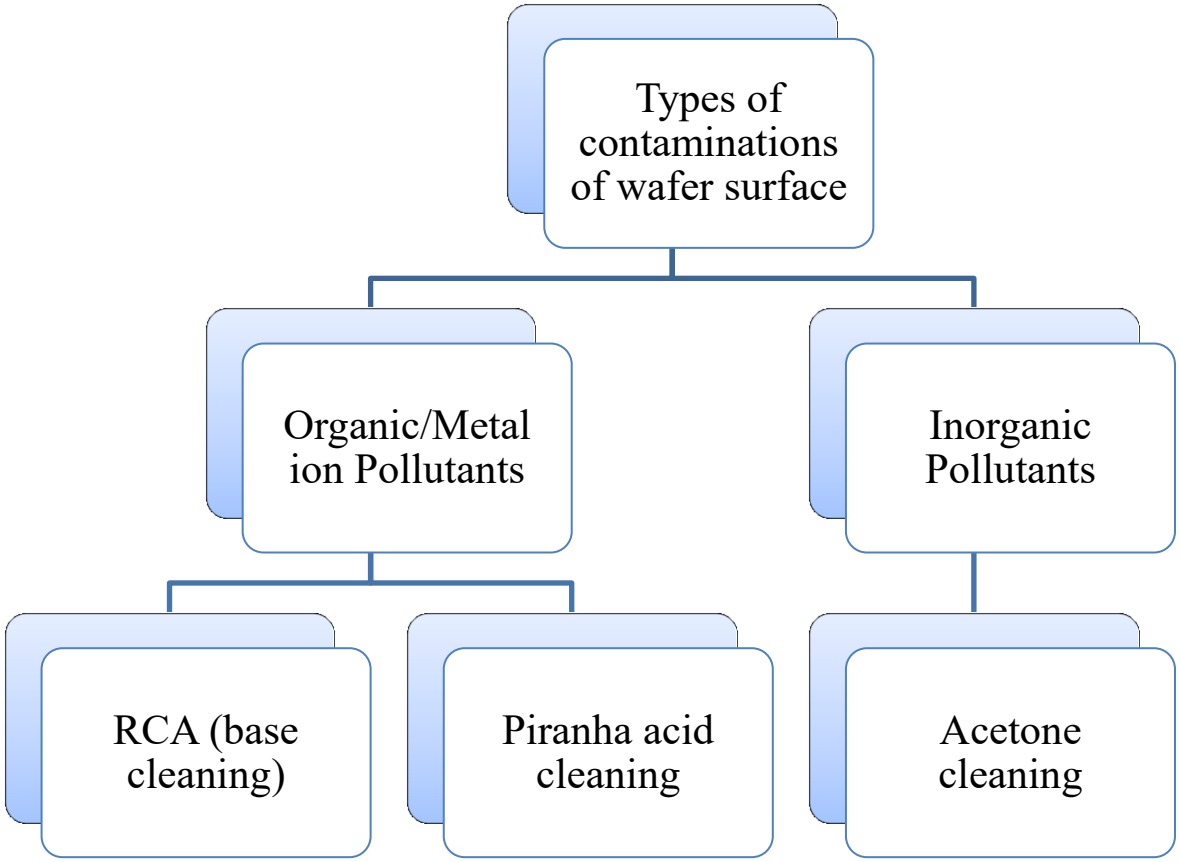


RCA: DI water+NH4OH+H202 (5:1:1)

Piranha: H202+H2SO4 (1:1 & 1:3) [This process is explained in Lab.]

Acetone: ACETONE+DI WATER

Some standard Silicon wafer cleaning processes

❑ Piranha Clean or Caro's acid clean or Sulfuric Peroxide Mix (SPM)

- Piranha clean or SPM is used to remove **organic** and **particulate contamination**.

- Treatment in freshly prepared H_2O_2 (30%) and H_2SO_4 (98%) in the ratio of 1:4 to 1:1 proportion for 30 minutes . The result of this mixture is formation of Caro's Acid

$$H_2SO_4 + H_2O_2 \longrightarrow H_2SO_5 \text{ (Caro's Acid)} + H_2O$$

(Caution: Add H_2SO_4 slowly into H_2O_2 not the reverse to avoid explosion)

The above process is an exothermic process as a result temperature rises up to 80°C - 120°C. So, all the operation is carried in a Quartz Beaker.

- Rinse in DI Water

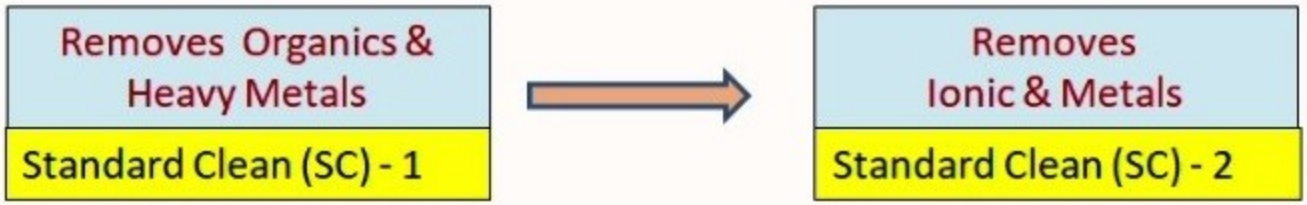
- Dip in 1-2% HF before subsequent processing to appear hydrophobia.

❑ RCA Clean

- Radio Corporation of America (RCA) cleaning process was first developed by Kern & Poutenin at RCA Laboratory, USA and was published in 1970. This process removes **organic, ionic** and heavy **metal contaminations.**

- It is mainly used by the Industries before fabrication of most critical steps.

- It consists of two sequential steps:



❖ SC-1 or Alkaline-Peroxide Mixture (APM)

- Treat the wafer in a freshly prepared solution consisting of **NH_4OH (27%), H_2O_2(30%)** and **H_2O** are in the ratio of **1:1:5** to **1:2:7** with temperature **70-80°C** is used for **10-15 minutes**.
- It doesn't provide self heat as like Piranha solution. So external heat is required (e.g. Hot plate or Bath).
- In this method **H_2O_2 oxidizes metals**, **NH_4OH** forms soluble complex with metal oxide (e.g. Ag, Cu, Zn, Cd, Au, Ni, Co & Cr) and OH ion cause removal of particles. Particles are removed due to modified zeta potential which is similar to how soap works to clean the surface.
- A surface chemical oxide (~15A) was formed in this process.
- **Dilute HF** (DHF) bath for **60 sec** is done at room temperature.

❖ SC-2 or Hydrochloric-Peroxide Mixture (HPM)

- Treat the wafer in a freshly prepared HCl (73%), H_2O_2(30%) and H_2O in the ratio of 1:1:6 to 1:2:8 at 70-80°C is used for 20 minutes.

- It also yields a thin chemical oxide (~15 A), which is etched in final HF dip.

- It is used to remove most metal contaminants & it also **removes alkaline ions like Na^+, K^+**

- In this method H_2O_2 oxidizes and HCl forms soluble chloride complexes with metal ions Na, Zn, Al, Mg, Fe etc.

DHF Clean / Oxide Etch

- It is used to remove the Chemical oxide formed on the surface of the Si wafer
- Dilute Hydrogen Fluoride (49%) and water in the ratio of 1:50 to 1:100 with room temperature is used for 01 to 02 minutes is used to remove the **native oxide layer** from the top of the surface of Si wafer.
- HF etches (Dissolves) SiO_2 and any **soluble particulates** embedded in the Oxide **float away**.
- After cleaning it leaves a **hydrophobic** (Water does not stick on the surface) clean surface. Si-H bond form on the top of the Si surface and surface become chemically inert (i.e. passivated surface). The passivation can last for minutes to hour.
- Oxygen from the ambient react with Si and form SiO_2 again.
- It can also cause some surface roughness, especially at high HF concentration and long time.

- **Rinsing**
  - Before and after every cleaning step Rinsing is done to remove the contamination.
  - This is the step actually cleans the surface by washing away the impurities.
  - All the chemistry, which we have done earlier, contamination still present in the solution. The way we remove the contamination is actually the rinsing.
  - Don't be Lazy in Rinsing
- **Blow Dry**
  - To dry the surface of the Si wafer, we are using blow dry using N_2 gun
  - It pushes the liquid off, without drying it.

Video

Specification of Si wafer

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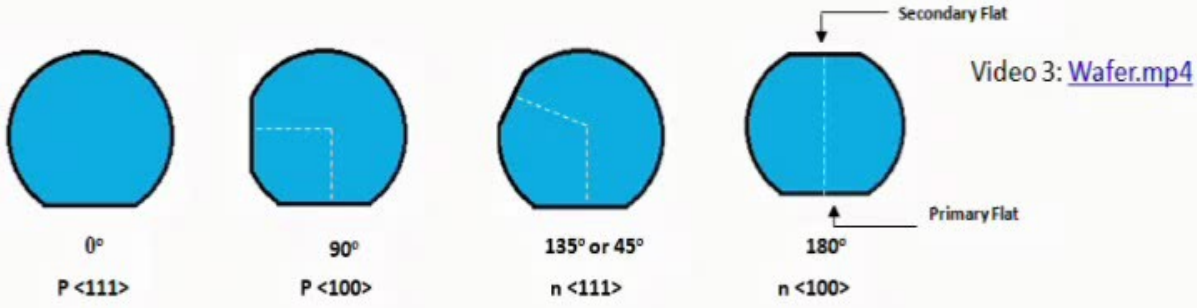
1.Jpg 2.jpg



Video 3: Wafer.mp4

Apparatus used: 1. Quartz beaker and wafer holder, 2. Teflon Tweezers,
3. Nitrile Gloves, 4. Borosil Glass beaker,
5. Nitrogen Gun, 6. Desiccator

Chemical Used: 1. 98% H_2SO_4, 2. 30%H_2O_2, 3. 1% buffer HF solution,
4. Nitrogen gas, De-ionised (DI) water (18.2 mega ohm-cm)

Procedure: 1. Place the contaminated silicon wafers in the quartz holder and put into the quartz beaker.

2. Poured H_2O_2 first and then poured equal amount of H_2SO_4 to maintain the ratio 1:1 [the order of mixing is not reversed to avoid exothermic explosion]
3. There is huge of amount of heat is evolved and bubbling is coming out which depicts the exothermic cleaning process. Kept the solution about 45 minutes.
4. The wafer is rinsed with DI water. The surface is now hydrophilic in nature due to presence of few nanometre thick native oxide layer.
5. The wafer again dipped into the 1% buffered HF solution to etch out the surface and remove the native oxide layer which is not required.
6. Again the silicon wafer is rinsed with DI water, the surface is now become totally hydrophobic. There was no water droplet is present on the surface.
7. This changes is the visual interpretation of removal of native oxide.
8. The wafer is dried with the help of N_2 gas and stored in a vacuum desiccator to avoid any further contamination.

Piranha process: $H_2SO_4+H_2O_2$—H_2SO_5 (Caro's acid)+H_2O

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Observation & Conclusion

- It is observed that if oxide is present on the surface of the wafer, the wafer develops a slight **pinkish tint**. If it does not have oxide deposition the wafer would look highly **shiny and silvery**.
- We have checked the Si Wafer before cleaning & after cleaning through Inverted Microscope. The silicon wafer was now cleaned and ready for further use.
- **Use only Quartz apparatus** since glass can be dissolved by the acid.
- The type of wafer that is to be used depends upon the utility and the purpose for which we want to fabricate the device. The rate of oxidations, efficiency, etc differs for different types of wafers. All these factors must be taken into account while choosing which type of wafer would be preferred for the required purpose.
- **Use Personal Protective Equipments** (PPE) and carefully handle the acid.
- **Never throw used chemicals down the drain.**